# Calcium sulphate filler composition


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| **Inventor:** | HEUSER GEOFFREY |
| **Applicant:** | POLYCELLS PRODUCTS LIMITED (GB) |
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Abstract of **GB2228931**

A filler composition, which composition when set has flexibility, contains (i) a hydraulically settable CaSO4, (ii) a flexibility-imparting amount of a polymer having a Tg temperature below 20 DEG C and (iii) water to set the settable CaSO4. The compositions of the invention are desirably in two part form and may employ the polymer in the form of an emulsion of MFT below 20 DEG C. The compositions may be used for woodwork repair.

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(71) Applicant
**Polycells Products Limited**

**(Incorporated in the United Kingdom)**

**Pentagon House, Sir Frank Whittle Road, Derby, DE2 4EE, United Kingdom**

(72) Inventor
**Geoffrey Heuser**

(74) Agent and/or Address for Service
**Boult Wade & Tennant**
**27 Furnival Street, London, EC4A 1PQ, United Kingdom**

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## (54) Calcium sulphate filler composition

(57) A filler composition, which composition when set has flexibility, contains (i) a hydraulically settable $CaSO_4$, (ii) a flexibility-imparting amount of a polymer having a Tg temperature below 20°C and (iii) water to set the settable $CaSO_4$. The compositions of the invention are desirably in two part form and may employ the polymer in the form of an emulsion of MFT below 20°C.

The compositions may be used for woodwork repair.

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# FILLER COMPOSITIONS

This invention relates to filler compositions and more especially to filler compositions which are particularly suitable for non-structural woodwork repair.

Filler compositions useful e.g. in non-structural woodwork repair (e.g. for restoring damaged window frames) should be flexible, allowing movement with the wood, e.g. during changes in temperature and humidity. The filler should also be non-shrinking so that on drying, or curing, the composition, cracking does not appear between the filler and the surrounding surfaces or in the filler composition itself. Furthermore, the filler composition should be easily applied and easy to shape after setting, for example by woodworking tools, glass paper or other sanding means. The composition, when set, should also be able to take varnish, woodstain or paint. Many of these requirements are generally useful in filler compositions regardless of application, particularly the possession of a degree of flexibility without detracting from strength in the cured product.

Filler compositions are well known in the art but in general do not meet the criteria necessary to provide a good flexible filler composition which is strong, and which can also be sanded or worked with cutting tools after curing.

For example filler compositions are known from GB2026003 comprising a particulate filler, such as silica, dolomite, or calcite and an aqueous binder resin emulsion. The resin emulsion comprises a quick drying polymer emulsion having an MFT above 5°C and

a flexible polymer emulsion having an MFT below 5°C. "MFT" is defined as the minimum temperature at which a polymer emulsion will form an integrated film. The MFT is relayed to the glass transition temperature, Tg, of the polymer, but also depends on other factors such as the polymer particle size in the overall emulsion and the surface tension of the overall emulsion. The glass transition temperature is the temperature at which a polymer loses its rubberiness or becomes crystalline as it is cooled.

Compositions comprising a hydraulically settable $CaSO_4$ (that is, a $CaSO_4$ material which can still take in water of crystallization, i.e. has less such water than the full dihydrate, gypsum) together with an aqueous resin emulsion are also known. They are not, however, fillers. Such compositions include the coating compositions of GB1390360 which are required to be fluid and "self-smoothing" but to "dry" to provide a "hard" inflexible coating, resistant to chipping and scratching, useful e.g. as a floor covering.

However, the art of filler compositions, despite a need for combinations of strength and workability and flexibility, does not contain any known compositions which are formulated to take advantage both of the strength of plaster whilst not losing the flexibility which certain polymers can provide in a cured composition. Filler compositions having both such ingredients are unknown.

Furthermore, the present invention (as will later be clear) provides a filler composition which cures through volume due to the $CaSO_4$ setting reaction in which water of crystallization is taken up (hydraulic

setting). This allows large repairs to be tackled with a single application. Water-based fillers known in the art dry by evaporation only, so they have to be applied in layers, allowing each layer to dry out before applying the next.

The composition of the invention also provides an advantage over known 2-part polyester based fillers: since they have low odour level, are non-toxic have a long working life, are non-slump and easy to mix, apply and sand. Polyester-based fillers have none of these advantages, and are both toxic and inflammable.

The present invention is based upon the discovery that non-toxic flexible but tough and workable fillers, notably wood fillers, can be provided by combining particular $CaSO_4$'s and polymers, preferably polymer emulsions, with care being taken over water content.

Thus, the present invention provides, a filler composition adapted to provide a cured filling which has flexibility comprising: (i) an hydraulically settable $CaSO_4$ present in an amount from 30 to 70% by weight of the composition, (ii) a polymer having a Tg temperature below 20°C present in an amount sufficient to impart flexibility to the composition when cured and (iii) water present in an amount not appreciably in excess of that sufficient to ensure hydraulic setting of the $CaSO_4$ and to permit mouldable paste formation; provided that if the amount of settable $CaSO_4$ is less than about 50% by weight an inert filler is also present. Such compositions are preferably, but not essentially, useable as wood fillers.

As used herein the term "mouldeable paste" is defined, with reference to the so called "Ball-Point" test, as the minimum amount of liquid required to convert a powder into a cohesive ball of paste.

The present compositions possess the necessary flexibility by virtue of the presence of a polymer having a Tg below 20°C. Examples of such polymers include a natural rubber latex or homopolymers or copolymers of ethylenically unsaturated monomers, e.g. olefins, such as ethylene, propylene; unsaturated carboxylic acids and alkyl esters thereof, e.g. acrylic, methacrylic, itaconic, fumaric and maleic acids, methyl acrylate, ethyl acrylate, n-butyl acrylate, methyl methacrylate, 2-ethyl hexyl acrylate, n-butyl methacrylate, di-n-butyl maleate, di-n-butyl fumarate, and n-lauryl methacrylate; vinyl and vinylidene halides, e.g. vinyl chloride and vinylidene chloride; vinyl esters of carboxylic acids, e.g. vinyl acetate, vinyl propionate, and the vinyl esters of mixed tertiary $C_{10}$ carboxylic acids; butadiene and isoprene; acrylonitrile and methacrylonitrile; and styrene and vinyl toluene, urethane or chloroprene, but other polymers will be suitable as long as they meet the Tg criterion.

As used herein, the term "flexible" is preferably but not essentially defined in terms of % extension at break for cast dumbells, of the material to be tested, when extended at a rate of 3.2 mm/minute on a Tensometer.

The compositions of the invention preferably have flexibility values which are greater than or equal to 1%, more preferably in the range from about 3 or 4% to about 20% and most preferably are about 4%.

It has been found that compositions comprising from 2 to 40%, preferably 5 to 35%, more preferably 10 to 30%, e.g. 15 to 25% by weight of polymer solids, and/or from 10 to 40% by weight of water, are especially suitable.

Hydraulically settable $CaSO_4$'s which may be used include, for example, anhydrous $CaSO_4$ or $CaSO_4$ hemihydrate, e.g. fine casting plaster or crystacal R (British Gypsum).

The compositions provided by the present invention exhibit the desired flexibility and are therefore useful in applications where such flexibility is desired, e.g. in the area of non-structural wood repair.

The invention also provides a two part filler composition comprising in the first part an hydraulically settable $CaSO_4$ and in the second part water, one or both of the said parts also including a polymer having a Tg temperature below 20°C; the said two parts being provided in sufficient amount to allow a single composition to be produced upon mixing in which the amount of the settable $CaSO_4$ is from 30 to 70% by weight, the amount of the polymer is sufficient to impart flexibility to the single composition after curing thereof, and there is sufficient water hydraulically to set the $CaSO_4$. It will be appreciated that a two part format is usually employed since the present compositions generally set within an hour.

Optionally the composition of the invention can further comprise an inert filler (e.g. Fillite) in an

amount up to 50% by weight of the composition. Generally, if the weight percentage of hydraulically settable $CaSO_4$ is less than about 50% then such an inert filler should be present in the composition. This increases the solid content of the composition and provides better sanding qualities.

Other components which can be included in the compositions are known in the art and include, inter alia, one or more of a reinforcing material, e.g. a mineral, vegetable or synthetic fibrous material, a dispersing agent, an antifoaming agent, a pigment, a thickening agent, e.g. cellulose ether or a polyacrylate, a set retarder, e.g. sodium Carboxy methyl cellulose, a set modifier, a waterproofing additive, e.g. calcium stearate, potassium methyl siliconate or a wax emulsion, an adhesion enhancer, e.g. polyvinyl, alcohol or a water reducing agent.

In highly preferred embodiments the invention provides a filler composition in which the polymer is provided in the form of an aqueous emulsion. Preferably the water content of the emulsion does not exceed the amount required hydraulically to set the $CaSO_4$ and to allow for mouldable paste formation. Such a polymer emulsion usually and preferably has an MFT below 20°C.

In a further preferred embodiment there is provided a wood filler composition which upon curing provides a filling which has flexibility consisting essentially of 59% by weight of a hydraulically settable $CaSO_4$, 11% by weight of a lightweight inert filler, 23% by weight of a polymer emulsion having an MFT below 20°C, 6% by weight of an adhesion enhancing polymer emulsion, and 1% by weight of a

water reducing agent. Preferably the polymer emulsion having an MFT below 20°C is a polyvinyl acetate copolymer emulsion and/or the adhesion enhancing polymer emulsion is a polyvinyl acetate homopolymer emulsion containing polyvinyl alcohol.

It will be appreciated that the order of mixing the various components is not essential to arrive at the final composition, with of course one proviso, i.e. that the hydraulically settable $CaSO_4$ component should not be brought into contact with the water until it is desired to produce the final composition since the $CaSO_4$ will begin to set upon mixing with the water.

One very important aspect of the invention is the use in a filler composition, which composition when set has flexibility, of (i) a hydraulically settable $CaSO_4$, (ii) a flexibility-imparting amount of a polymer having a Tg temperature below 20°C and (iii) water to set the settable $CaSO_4$. As previously indicated, the known art does not contain any such technique for fillers. By using this technique a filler paste can be produced which, in prefered embodiments, remains workable for 15 to 25 minutes (e.g. 20 minutes) and sets within 30 to 40 minutes to a hard, water resistant solid which may be sanded to a smooth finish and dyed, stained, painted or varnished.

The preferred wood fillers of this invention are flexible so as to move with the natural movement of wood under different conditions, e.g. as the weather may be alternately wet and dry.

The present invention further provides, in other aspects, inter alia,

1) A filler pack containing essentially hydraulically settable $CaSO_4$ and a polymer having a Tg temperature below 20°C such that when water is added a composition of the invention results;

2) A filler kit which contains two packs, a first pack containing a hydraulically settable $CaSO_4$ and a second pack containing an aqueous emulsion of a polymer having a minimum film forming temperature below 20°C, the contents of the packs being such that when they are mixed there is provided a composition of the invention; and

3) A method of filling, optionally filling wood, which comprises loading into the space to be filled a composition (or ingredients thereof) comprising (i) an hydraulically settable $CaSO_4$ present in an amount from 30 to 70% by weight of the composition, (ii) a polymer having a Tg temperature below 20°C present in an amount sufficient to impart flexibility to the composition when cured and (iii) water present in an amount not appreciably in excess of that sufficient to ensure hydraulic setting of the $CaSO_4$ and to permit mouldable paste formation; provided that if the amount of settable $CaSO_4$ is less than about 50% by weight an inert filler is also present.

The invention will now be further described with reference to the following Examples. It should be

noted that although the filler compositions described in the Examples are for use as wood fillers, this should not be taken to represent a limitation of the overall invention.

In relation to the compositions of the Examples, the following tests were carried out and the results appear in the Table.

1. Setting time:- determined using a Vicat setting time apparatus;

2. Flexibility:- determined by casting dumbells of the material in silicone rubber mould taking care to achieve a smooth surface, they are then demoulded after 24 hours at room temperature and lines are carefully marked in pencil on a dry face to show a 2 inch length. The dumbells are then left to dry for 3 days and are then extended to break at a rate of 3.2 mm/minute on a J.J. Lloyd Tensometer Model T22K;

3. Adhesion:- measured by the SATTEC pull off test. The material is applied to a blockboard using a template as a guide, is allowed to dry for 48 hours before Al.SATTEC discs are stuck on using epoxy. After a further 24 hours the Al discs are pulled using the SATTEC apparatus. The reading in Newtons is noted and the type of failure of the product;

4. Dynamic performance:- measured by filling 25 mm holes in soft wood with the

composition and subjecting the wood to dry and wet cycles in a J.B. Marr Weatherometer.

5. Ease of sanding, ability to take paint, varnish and woodstain.

Example 1

Part A

| | | % by weight |
|---|---|---|
| Fine Casting Powder | (Hemihydrate plaster) | 54.9 |
| Arbocell | (Woodfibre for reinforcement) | 10.0 |
| Polyvinyl Alcohol | (Adhesion additive) | 5.0 |
| Cellulose ether | (Thickener) | 0.1 |

Part B

| | | |
|---|---|---|
| Styrene/Acrylic emulsion | | 30 |

Example 2

Part A

| | | % by weight |
|---|---|---|
| Fine Casting Plaster | | 61.7 |
| Powder polyacrylate | (Thickener) | 1.3 |

Part B

| | | |
|---|---|---|
| Alkyd emulsion | | 18.5 |
| Water | | 18.5 |

Example 3

Part A

| | % by weight |
|---|---|
| Crystacal Plaster | 58.75 |
| Lightweight filler | 15.0 |
| Polyester fibre | 1.0 |
| Polyvinyl alcohol | 0.2 |
| SCMC retarder | 0.05 |

Part B

| | |
|---|---|
| Styrene/butadiene latex | 20.0 |
| Water | 5.0 |

Example 4

Part A

| | % by weight |
|---|---|
| Fine Casting Powder | 39.3 |
| Wood fibre | 7.2 |
| Polyvinyl alcohol | 3.6 |
| Cellulose ether | 0.1 |
| Powdered Polymer | 30.0 |

Part B

| | |
|---|---|
| Water | 28.5 |

Example 5

Part A

| | % by weight |
|---|---|
| Crystocal R Plaster | 59 |
| Lightweight filler | 11 |

Part B

| | |
|---|---|
| Polyvinyl Acetate copolymer emulsion | 23 |
| Polyvinyl Alcohol homopolymer emulsion/ Polyvinyl Alcohol | 6 |
| Water reducing agent | 1 |

TABLE

| Formulation Number | Setting Time (minutes) | Flexibility (% extension at break) | Adhesion SATTEC ($KN/m^2$) | Dynamic performance (3 cycles) | Ease of sanding | Ability to take paints, varnish and woodstain |
|---|---|---|---|---|---|---|
| 1 | 45 | 3 | 1137 | good performance except for blistering | easy to sand | good |
| 2 | 27 | 1 | not tested | peripheral cracking | very easy to sand | good |
| 3 | 14 | 1 | not tested | peripheral cracking | easy to sand | good |
| 4 | 60 | 22 | 874 | slight swelling | more difficult to sand | good |
| 5 | Satisfactory | 4 | 551 | no failure | easy to sand | good |

CLAIMS:

1. A filler composition adapted to provide a cured filling which has flexibility comprising: (i) an hydraulically settable $CaSO_4$ present in an amount from 30 to 70% by weight of the composition, (ii) a polymer having a Tg temperature below 20°C present in an amount sufficient to impart flexibility to the composition when cured and (iii) water present in an amount not appreciably in excess of that sufficient to ensure hydraulic setting of the $CaSO_4$ and to permit mouldable paste formation; provided that if the amount of settable $CaSO_4$ is less than about 50% by weight an inert filler is also present.

2. A composition as claimed in claim 1 which comprises an inert filler in an amount of up to 50% by weight of the composition.

3. A composition as claimed in claim 1 or claim 2 which comprises from 2 to 40% by weight of polymer solids and from 10 to 40% by weight water.

4. A composition as claimed in any one of claims 1 to 3 wherein the polymer is a natural rubber latex or a homopolymer or copolymer of ethylene, propylene, acrylic acid, methacrylic acid, itaconic acid, fumaric acid, maleic acid, methyl acrylate, ethyl acrylate, n-butyl acrylate, methyl methacrylate, 2-ethyl hexyl acrylate, n-butyl methacrylate, di-n-butyl maleate, di-n-butyl fumarate, and n-lauryl methacrylate, vinyl, vinyl chloride, vinylidene chloride, vinyl acetate, vinyl propionate, butadiene, isoprene, acrylonitrile and methacrylonitrile, styrene, vinyl toluene, urethane or chloroprene.

5. A composition as claimed in any one of claims 1 to 4 further comprising one or more of a reinforcing material, a dispersing agent, an antifoaming agent, a pigment, a thickening agent, a set retarder, a set modifier, a waterproofing additive, an adhesion enhancer or a water reducing agent.

6. A composition as claimed in claim 5 wherein the waterproofing additive is calcium stearate, potassium methyl siliconate or a wax emulsion.

7. A composition as claimed in claim 5 or claim 6 wherein the thickening agent is a cellulose ether or a polyacrylate.

8. A composition as claimed in any one of claims 5 to 7 wherein the adhesion enhancer is polyvinyl alcohol.

9. A composition as claimed in any one of claims 5 to 8 wherein the reinforcing material is a mineral, vegetable or synthetic fibrous material.

10. A composition as claimed in any one of claims 5 to 9 wherein the set retarder is sodium Carboxy methyl cellulose.

11. A composition as claimed in any one of claims 1 to 10 wherein the polymer is provided in the form of an aqueous emulsion.

12. A composition as claimed in claim 11 wherein the water content of the emulsion does not exceed the amount required hydraulically to set the $CaSO_4$ and to permit mouldable paste formation.

13. A composition as claimed in claim 11 or claim 12 wherein the emulsion has an MFT below 20°C.

14. A composition as claimed in any one of claims 1 to 13 wherein the hydraulically settable $CaSO_4$ is anhydrous $CaSO_4$ or $CaSO_4$ hemihydrate.

15. A composition as claimed in any one of claims 1 to 14 which is a wood filler composition.

16. A two part filler composition comprising in the first part an hydraulically settable $CaSO_4$ and in the second part water, one or both of the said parts also including a polymer having a Tg temperature below 20°C; the said two parts being provided in sufficient amount to allow a single composition to be produced upon mixing in which the amount of the settable $CaSO_4$ is from 30 to 70% by weight, the amount of the polymer is sufficient to impart flexibility to the single composition after curing thereof, and there is sufficient water hydraulically to set the $CaSO_4$.

17. A two-part filler composition as claimed in claim 16 and as further defined by the specific feature(s) of any one or more of claims 2 to 15.

18. A wood filler composition which upon curing provides a filling which has flexibility consisting essentially of 59% by weight of a hydraulically settable $CaSO_4$, 11% by weight of a lightweight inert filler, 23% by weight of a polymer emulsion having an MFT below 20°C, 6% by weight of an adhesion enhancing polymer emulsion, and 1% by weight of a water reducing agent.

19. A wood filler composition as claimed in claim 18 wherein the polymer emulsion having an MFT below 20°C is a polyvinyl acetate copolymer emulsion and/or the adhesion enhancing polymer emulsion is a polyvinyl acetate homopolymer emulsion containing polyvinyl alcohol and/or the water reducing agent is a melamine/formaldehyde condensate resin.

20. The use in a filler composition, which composition when set has flexibility, of (i) a hydraulically settable $CaSO_4$, (ii) a flexibility-imparting amount of a polymer having a Tg temperature below 20°C and (iii) water to set the settable $CaSO_4$.

21. A filler pack containing essentially hydraulically settable $CaSO_4$ and a polymer having a Tg temperature below 20°C such that when water is added a composition as claimed in any one of claims 1 to 15 results.

22. A filler kit which contains two packs, a first pack containing a hydraulically settable $CaSO_4$ and a second pack containing an aqueous emulsion of a polymer having a minimum film forming temperature below 20°C, the contents of the packs being such that when they are mixed there is provided a composition as claimed in claim 13.

23. A method of filling, optionally filling wood, which comprises loading into the space to be filled a composition (or ingredients thereof) comprising (i) an hydraulically settable $CaSO_4$ present in an amount from 30 to 70% by weight of the composition, (ii) a polymer having a Tg temperature below 20°C present in an amount sufficient to

impart flexibility to the composition when cured and (iii) water present in an amount not appreciably in excess of that sufficient to ensure hydraulic setting of the $CaSO_4$ and to permit mouldable paste formation; provided that if the amount of settable $CaSO_4$ is less than about 50% by weight an inert filler is also present.

24. A composition as defined in claim 1 and substantially as hereinbefore described.

25. A composition substantially as hereinbefore described in any one of Examples 1 to 5.

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